

HANDBOOK OF IMAGING MATERIALS

EDITED BY

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Preface

The field of imaging technology has undergone massive changes over the past fifty years, from the 1940s, when photography served the entire gamut of reprographic applications, to its narrower focus in the imaging industry today. A half-century ago, silver halide-based papers and films performed every task from image capture to document copying.

Diazo processes took on special importance during the war years in the 1940s, growing rapidly in the 1950s to become the predominant method for engineering drawing reproduction. Electrophotography grabbed the spotlight in the late 1950s and in the 1960s, when it revolutionized office procedures by increasing worker productivity through automation. Exploitation of this technology continues apace in facsimile, in desktop publishing, and in other areas of business communication.

Today, silver halide emulsion is still the primary image-capture medium for visible light, infrared, and x-ray photography. Amateur photographers, carrying some 250 million 35mm cameras worldwide, depend upon this technology. In the United States alone, the retail photofinishing market reported revenues of \$5.34 billion in 1989, based on almost 15 billion exposures on silver film.

Although it is now a commercial reality, the digital, electronic camera is not expected to equal the number of photographs taken on silver film until the turn of the century, when an estimated 23 billion exposures will be made in this country.

While silver photography continues to hold on to the most demanding

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5.3.2 Magnetic Materials Required

As stated previously, all the materials mentioned can be adapted to cascade development. All that is required is to design a mechanism that will move and mix the developer. In the past 20 years the magnetic brush development has restricted the choices to "soft" magnetic materials; that is, the material must be magnetically soft or have very low loss and low remanence. Once a material has been in a magnetic field, it retains very little polarization when removed from the field. If the remanence is high, each bead becomes a tiny permanent magnet and all the beads attract one another, severely restricting the flow properties upon returning to the sump for remixing. The "soft" magnetic materials used are iron, low carbon steels, annealed nickel, magnetite, and ferrites. Recently, Eastman Kodak has broken new ground by introducing "hard" ferrite carrier into some of their machines. This material has some of the problems associated with flow after magnetization, but Eastman has designed some ingenious developer housings to address the situation. The "hard" materials have considerable remanence and are generally used in permanent magnet applications such as the novelty refrigerator magnets.

The need for cleaner, sharper, high quality copies full of solid areas for brochures, graphs, and diagrams has forced many improvements in the carrier materials. The ultimate goal is to have copies that compare to lithography for resolution, solid fill, and uniformity. This requires a controlled and consistent resistivity of the developer, generally lower than the insulating sand or glass and higher than the iron or steel. Therefore most of the iron powders are oxidized to control the resistivity and partially coated to control the electrostatic charging. Irregular iron powders have been used in the past few years, and the theory is basically that the high points are oxidized⁷ and poorly coated, to supply the required resistivity, while the valleys are better coated, to supply the charging effect required.

Many of the Japanese copiers used irregular iron such as flake or filings. In recent years most of the two-component copiers have shifted to ferrites because of their semiconducting properties. These materials have resistivities in the range desired because they are transition metal oxides and magnetic ceramic materials, which in some applications can be used without partial coating for toner charging.

5.4 CURRENT CARRIER POWDERS

5.4.1 Steel (Spherical)

One of the first uses of 450 μm spherical steel came in 1969 with the introduction by Xerox Corporation of a 60 copy per minute machine called the 3600-III. The process for materials of these types is described in patents by R. Hagenbach and R. Forgesi for classifying and manufacturing by a two-wire process.^{8,9}

In the same time frame, development of rotating electrode process was going on at Nuclear Metals, Inc. This process (Fig. 5.4) took advantage of one of the first materials designed specifically for carrier powders. Another highly successful machine, the 3100, was introduced by Xerox in 1972. This 12 copy per minute machine, which utilized 100 μm spherical steel as the carrier with a magnetic brush developer housing, led to a series of similar machines, all using the same developer. Many of them are still in service. In 1971 IBM entered the copier field with the introduction of the Copier I, which used Teflon-coated 170 μm spherical steel. IBM continued to use this material in most of their subsequent machines. Since all these copiers used coated carrier, the developer was quite insulating; consequently, the solid area reproduction was poor, and background and machine dirt have been the source of problems.

Table 5.1 also lists some other applications of spherical carriers for reference.

5.4.2 Iron (Irregular)

Irregular iron was first demonstrated by Greig, Giaimo, and Young with the invention of Electrofax at RCA for a coated paper copier. This became a commercial reality in 1961 when it was marketed by the American Photocopy Equipment Company as "DriStat." The next important entry of irregular iron came in 1975, when Eastman Kodak entered the copier field with the Ektaprint 100/150.¹⁰ This machine used a coated 170 μm carrier and made use of the resistivity and charge controls by oxidation⁷ and coating as described previously. This material is a reduced screened magnetite, which results in a very rough bead of nearly pure iron.

The same type of material is utilized in the Xerox 1075, which came on the market in 1983. The size is slightly smaller at 130 μm , but the same resistivity and charge control processes are applied. A different 100 μm irregular iron made by water atomization was introduced in 1980 by Xerox Corporation in a machine called the 3300. This carrier is oxidized and coated similarly for the same reasons.

Recently a partially reduced magnetite has been used in some Japanese copy machines and is described in a joint patent between Kanto Denka Kogyo in Japan and Hoegaans AB in Sweden.¹¹ All these machines reproduce solid areas quite well and deliver excellent copy quality.

5.4.3 Soft Ferrites (Spherical)

The simple ferrite process flowsheet illustrated in Figure 5.5 includes the spray drying step, which is where the spherical powder is formed. Many ferrite formulations may be made in this process and adapted to the application of carriers for xerography. The first mention of ferrites to be used in magnetic brush development was by Joseph Wilson of Haloid-Xerox, Inc. in 1958.¹² The first

commercial use came in 1975. This family of materials has grown steadily in the past 15 years from the first introduction in a high speed (120 copies/min) machine called the 9200 from Xerox Corporation.¹³⁻¹⁷ Since that time ferrites have been used in more than 20 Xerox copiers.

The trend to ferrites spread to the Japanese copier companies, which in 1986 alone introduced 38 new copiers and printers, of which 34 utilized ferrite carrier. These materials have several advantages in magnetic brush development that allow them to be tailored to the development housing design and the charging properties required for any given machine. The saturation magnetic moment can be varied by formulation and process from 20 to 75 electromagnetic units per gram, which relates to the developer pickup on the brush and the stiffness of the brush. Too high a moment will result in a stiff brush, which will develop but will also scratch the image, causing nonuniformities in the solids and ragged edges on the line copy or letters. In the case of iron or steel, the saturation moments range from 150 to 190 emu/g and cannot be varied much. Ferrites are lower density materials and therefore require lower torque to drive the developer housing, resulting in lower energy input requirements. Ferrites are semiconductors naturally, and the resistivity can be varied across the useful range of carriers, which is generally 10^6 – $10^{12} \Omega \cdot \text{cm}$. They have been used in some cases without the requirement of partial coating. The surface microroughness can be controlled so that the material is consistently coatable in any given coating process that it might require. The size range is variable, from 10–120 μm for the spherical ferrites down to 2 μm for irregular ferrites, which are starting to raise some interest in the industry.¹⁸

5.4.4 Hard Ferrites (Spherical)

The commercial use of the hard or permanent magnet type of ferrite is a very recent development. In 1988 Eastman Kodak announced the availability of the Coloredge, which uses a smaller carrier that is a hard ferrite and is described in a patent by Miskinis et al.¹⁹ This is a high speed (23 copies/min), full color copier, which can give color highlights at higher speeds. This machine has multiple developer housings to accommodate the primary colors for a full spectrum output.

5.4.5 Hybrid Mixtures

Another earlier full color copier that used multiple housings was the Xerox 6500, introduced before its time in 1973, when there were not enough color originals for it to copy. Now that full color printers are flourishing, there is an explosion of interest in this area. The 6500 is included in the hybrid category because it used two different carriers. One color utilized 100 μm nickel-beryllium, which is an electrolytically grown nodule of high purity nickel. Because of the expense and

sometimes wild fluctuations in price, as well as military priorities, this material has not been used in other applications to this writer's knowledge.

In another hybrid ferrite composition, developed in 1986 by Hitachi Metals, Ltd., two different ferrite crystalline structures are mixed in the standard ferrite process to make a copier powder²⁰ that is used in some of the Japanese machines. The composition is a mixture of a hexagonal "hard" ferrite and a spinel "soft" ferrite; and although it is not stated in the disclosure, this is probably a two-phase core with the hard phase having little effect in the low magnetic fields experienced in the magnetic roll of the machine, hence having little magnetization effect.

Still another hybrid is a mixture of two or more of the materials already mentioned. An example of this is a mixture of spherical ferrite and iron flake noted in at least one of the Panasonic copiers. There is also a patent by Cooper and Goldstein that describes a similar mixture.²¹

5.5 CARRIER MANUFACTURING PROCESS

This section describes some of the methods used to convert the raw powder into carrier. Many carriers are coated or, more often, precisely partially coated for two main reasons. One is to enhance the toner charging ability, because polymer surfaces give better charge exchange and less charge recombination than the inorganic surfaces. The other reason is that the adhesion forces or "stickiness" is less with polymer surfaces, and there is less toner adhered to the carrier surface, an effect that impedes development and shortens the life of the carrier. When toner is adhered or too highly charged, it uses up carrier area until there is not enough charging surface left to support the toner required to give the image density expected. Any further toner additions result in low or no charging, and an increase in background and machine dirt. Such cases of developer failure generally require replacement of the developer charge and a costly maintenance call to replenish the machine.

Several investigators have generated a triboelectric series by rubbing different surfaces together and determining the charge exchange. The series is arranged to show that a material will acquire a positive charge from the material just below it in the series and a negative charge from the material just above it. The following short list is an example.²²

- | | |
|--------------------------|-------------------------------------|
| 1. Air | 8. Polymethyl methacrylate |
| 2. Glass | 9. Polyester |
| 3. Nylon, wool, and silk | 10. Polyurethane |
| 4. Aluminum | 11. Polystyrene |
| 5. Cellulose | 12. Polyethylene |
| 6. Cellulose acetate | 13. Polypropylene |
| 7. Iron | 14. Polytetrafluorethylene (Teflon) |

Many variables (moisture, impurities, etc.) have effects on the charging properties. This is why there are some differences in the triboelectric series from different sources.²² The list of polymeric coatings is too long to include here, but most of the carrier patents listed in the references for this chapter provide a short history covering an extensive list of materials that have been used.

The majority of the carriers in production today are coated by one of the two methods discussed next. Another minority group uses uncoated carrier, which is just the raw powder. Still another small percentage use an oxidized iron core with no coating, and this will be briefly covered.

5.5.1 Solvent Coating

One of the most common methods of coating carrier is solvent coating. There are several types of equipment in use today, ranging from the very simple to the ultrasophisticated. For example, it might be desired to coat polymethyl methacrylate (PMMA) from a solution in toluene. One method involves the use of a vibratory tub with a cover connected to a suction fan to pull the solvent vapors through a cooling unit to recover the toluene. The powder can be preheated and placed in the unit, or some vibratory coaters can be constructed with a steam-heated jacket. After the powder has been heated to a specific temperature, the solution of PMMA in toluene (generally 10–15% PMMA by weight) is added slowly by spraying onto the powder. The heat and the vacuum created serve to evaporate the solvent, leaving the polymer on the surface of the beads. The tub has a curved bottom, and the constant vibration keeps the powder flowing in one direction and turning over on itself. Generally a stirring bar is required to break up the agglomerates as the coating goes through the tacky phase. The solution is added until the precalculated coating weight is achieved. Agitation and heating are continued until the bed of powder starts to rise in temperature, indicating that the solvent is gone. The agitation continues until the temperature drops to a level for safe removal of the coated product.

An example of the more sophisticated coater is the use of a twin-cone vacuum drier with a heated shell and metered solution input. The unit is connected to a vacuum pump, which pulls the solvent vapors through a condenser to recover the solvent. Various types of this apparatus exist today, and some have feedback controls that vary the vacuum and solution feed rate to control the temperature through the coating cycle. In this case the powder is placed in the unit at ambient temperature and heated to the initiation temperature, at which time the addition of the solution is started, the rest of the process is similar to the vibratory tub. This procedure gives generally a more consistent coating, but it is important to note that most powders require a partial coating and different methods of coating will give different toner charging results even at the same coating weight. Solvent coaters of several other types are in use, but the principles are the same and the results must be tuned to the application in a specific machine.

5.5.2 Powder Coating

A second extensively utilized coating method is powder coating. That is, the coating is applied as a dry, very fine powder such as polyvinylidene fluoride (PVF). In this case the PVF ($< 5 \mu\text{m}$) is added to the core at the desired coating weight and tumbled until the PVF has adhered to the core by adhesion and/or electrostatic forces. The material is then subjected to a temperature high enough to melt the polymer and spread over the core surface. This is generally done in a rotary tube furnace, so that the powder rolls over itself and progresses down the inclined tube. The output is a coated carrier, but again the process parameters must be tuned to obtain the desired toner charging results for a specific application. This type of coating is mainly used for the irregular powder aimed at controlling resistivity and charging response.

5.5.3 Oxidation

The oxidation method of surface treatment applies mainly to iron and steel powder, since the ferrites are all oxide already. The process forms a ferritlike oxide surface and raises the resistivity of the base material. It also prepares the surface for better coating adhesion, just as most metal surfaces are prepared for painting or coating. One illustrative method of this is described in a patent cited earlier.⁷ In this case the powder is fluidized in a cylindrical container and the fluidizing air is heated to a specific temperature for a controlled length of time and then cooled in a controlled atmosphere to produce the fine-grained type of surface oxide required. Often this process is targeted to give a specific color, because the various oxides of iron range from red to yellow to dark blue depending on the temperature and time involved. Of course, the qualifying parameter is ultimately the resistivity of the powder. It may or may not be necessary to coat this product, depending on the application.

Coating is a batch process, and many companies achieve the same end by using a rotary tube furnace to oxidize their powder. This is a little more tricky because the temperatures required is generally just below one of the phase transition temperatures of iron oxide and the phase change is exothermic; thus if the temperature is exceeded, a runaway reaction with localized melting may result. This at best stops the process and at the worst damages the furnace.

5.6 CARRIER PROPERTIES

5.6.1 Magnetic Properties and Testing

The magnetic characterization of carrier powders is important, as are all the properties to follow, in obtaining a controlled product in a consistent manner. In other words, the specification agreement between vendor and customer is a

critical document. Most users of these materials require two and sometimes three points on the direct current hysteresis loop.

This is a test that subjects the powder to a slowly increasing magnetic field and measures the magnetization of the sample in the field. The magnetic field is increased to saturate the material, reduced to zero, reversed to saturate the sample in the opposite sense, and reduced again to zero. The sample magnetization is plotted as a function of the magnetic field applied; the hysteresis loop that results characterizes the sample. In the stylized loops illustrated in Figure 5.6, note especially the soft and hard types of magnetic material that have been discussed.

One piece of the equipment for obtaining such data (Fig. 5.7) is called a vibrating sample magnetometer (VSM). This device was developed by S. Foner in the early 1960s and has been sold commercially since that time by Princeton Applied Research. The principle of operation is simply that the sample is vibrated in a vertical mode and the magnetic field of the electromagnet is applied orthogonal to the motion. Since the magnetization of the sample is the only parameter varying with time, it is picked up by the sensing coils, integrated electronically, and displayed as a function of the applied field. The VSM is

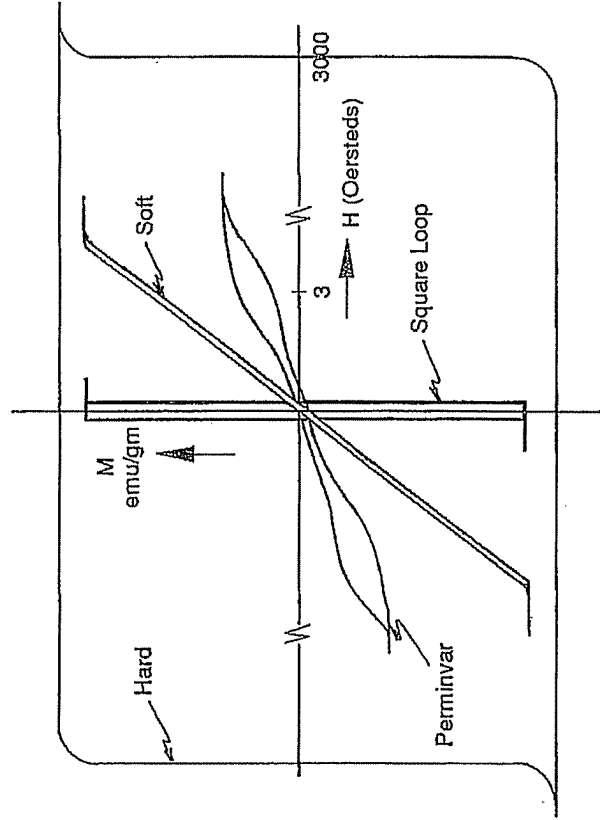


Figure 5.6 Generic hysteresis loops: M , moment; H , force.

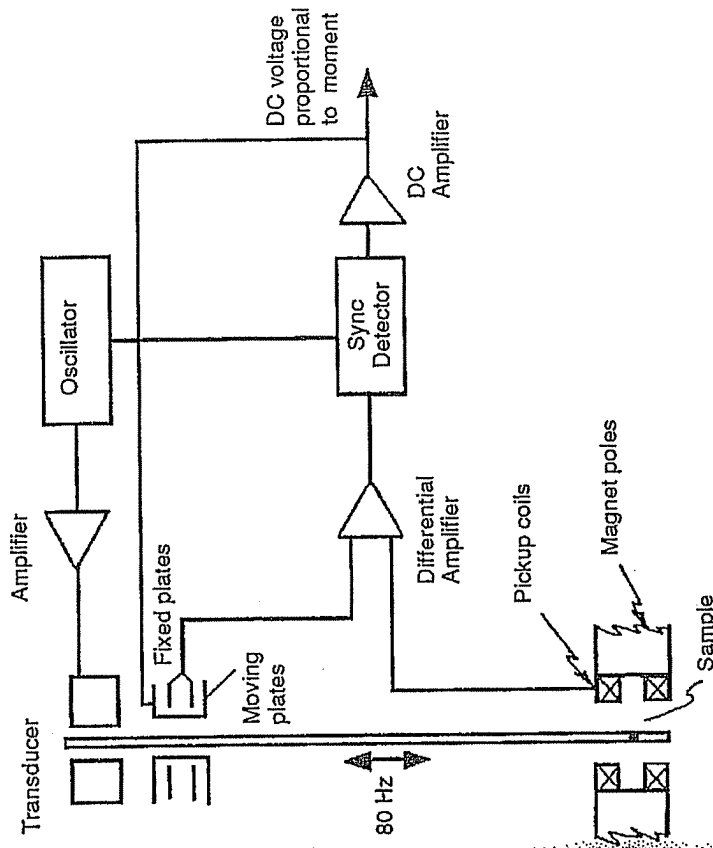


Figure 5.7 Vibrating sample magnetometer schematic.

calibrated using 99.999% pure nickel, which has a saturation moment of 55.01 emu/g. This calibrates the Y -axis for electromagnetic units, and the sample value is divided by its weight, resulting in a traceable, precise, and accurate measurement. The voltage on the coils is:

$$E = N \frac{d\phi}{dt}$$

where N is the number of turns, ϕ is the flux from the sample, and t is time.

Integration with respect to time gives:

$$E(\phi) = \frac{Et}{N} (V \cdot s)$$

but this output voltage is calibrated with the standard in electromagnetic units, so that the sample can be read directly in those units. A complete hysteresis loop

can be displayed on an x-y plotter and will resemble the curves shown in Figure 5.6.

Recently a more compact and less expensive unit that does the same thing, called an alternating gradient magnetometer (AGM) has been offered by Princeton Measurements Corporation.²³ There are single-point measurement units that will look at just the saturation magnetization and/or the remanence magnetization. From the xerographer's point of view, these values are useful. The saturation moment (M_s) can be correlated to the stiffness of the brush in any given machine. The remanence moment (M_r) can be correlated to the flow of material in the sump during mixing. The coercive force (H_c) or the magnetic field required to reduce the remanence to zero is a measure of the difficulty in magnetizing the material or the "softness." The higher the H_c , the harder the material.

5.6.2 Electrical Properties and Testing

The electrical properties of powders are complex because the materials are solid but act like liquids. A myriad of methods have been developed to obtain a number representing the resistivity of these materials. Most xerographic engineers like to measure the powder in a cell that "simulates" the magnetic brush. Thus most companies have their own cell that looks something like Figure 5.8, with a miniature magnet roll, power supply, and meters. Some measure with the brush moving and some form the brush and measure with it stopped. Many different voltages are used, and the calculations of resistivity all assume some brush cross-sectional area.

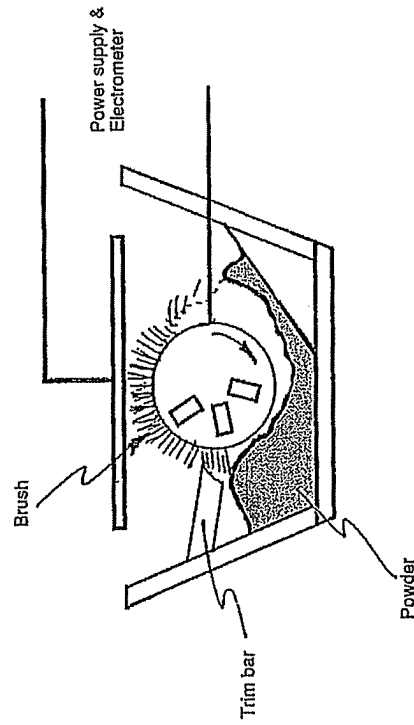


Figure 5.8 Generic dynamic cell.

There are several problems with this method. First, it is very difficult to build two cells to be identical so that people can make the measurements in different locations, and this is a disadvantage for vendor-customer correlation. Second, there are no standards for powder resistivity, so the parties must pick variations in the material, establish standards, and negotiate the values by agreement. Finally, since there is no industry standard cell available, cells must be built by the interested parties.

Even though there is no standard material for reference, the more precise measurement that can be correlated is possible on a static cell (Fig. 5.9). This liquid dielectric cell uses about a pound of material; it is easily cleaned, and the micrometer adjustment gives accurate spacing. The cell is simply a guarded electrode test for the resistivity or dielectric properties of liquids. The powder is assumed to be a liquid, and a voltage is applied across the sample while measuring the current. The area of the inner electrode is used in calculations. The measurement is repeatable, and correlation is good between locations. This cell is an old standard in the industry and is available to everyone.

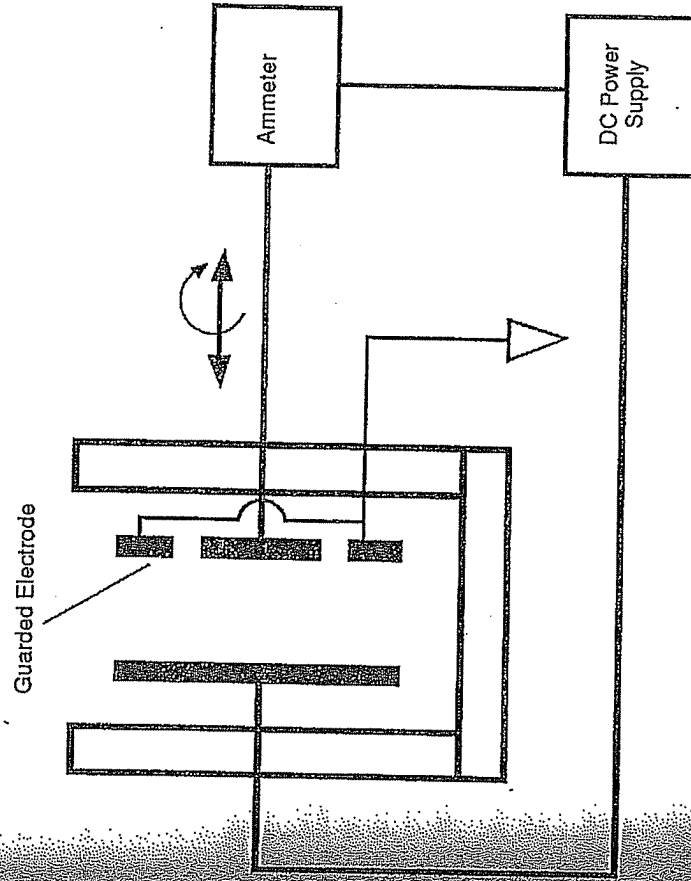


Figure 5.9 Static resistivity cell.